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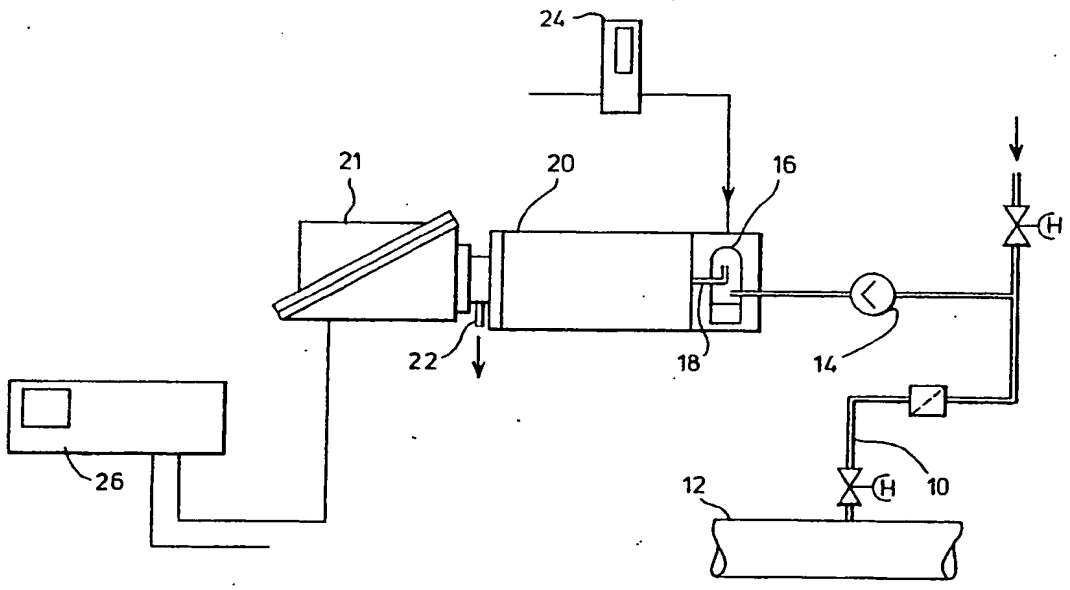

(57) Abstract

Method and apparatus for analysis of liquids such as beer comprises an evaporator to introduce the liquid to be analysed into the vapour phase, and near infrared analysis apparatus to determine the solute content of the liquids. By the use of vapour phase analysis, the analytical performance of the apparatus with respect to the content of liquid such as alcohol is greatly improved.

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METHOD AND APPARATUS FOR ANALYSING LIQUIDS

This invention relates to a method and apparatus for analysing liquids. A particular example of the application of the invention is to the analysis of the alcohol content of liquids such as beer, wine etc. The invention may also find application to analysis of the solute content of other solutions, such as ammonia in water.

The brewing industry requires accurate methods of measuring the alcohol content of beer produced, as duty is charged on beer on the basis of the alcohol content. Furthermore, the recent increase in the popularity of "low-alcohol" and "no-alcohol" beers has lead to a need for accurate control of the alcohol content at very low alcohol levels. Accordingly, the industry needs to be able to measure alcohol content at levels as low as, for example, 0.5%, and with an accuracy of 0.07%.

Previous proposals for the measurement of alcohol content in beer have included the use of near infra-red analysis apparatus employing transmission cells containing the beer to be analysed. Such methods are however mathematically complex and do not provide the required level of accuracy. They stretch the technology of near infra-red liquid analysis to its limits, and are almost certainly not adequate for the measurement of alcohol content in low-alcohol beers. A further factor is that this approach to the analysis is inevitably laboratory-based. Nevertheless, to-date it has been long accepted that this is the best that can be done.

The requirements of the brewing industry as discussed above include a need for apparatus and a method capable of providing substantially continuous monitoring of alcohol content, including measurement at the low levels of alcohol mentioned above, which are applicable to the production of beer on a continuous basis in an industrial plant.

Accordingly, we have identified a requirement for improvements in relation to the analysis of liquid solutions,

particularly in relation to liquids such as beer, wine etc, offering improvements in relation to matters discussed above, or generally.

According to the invention there is provided a method and apparatus for analysis of liquid solutions as defined in the accompanying claims.

In a preferred embodiment, the method and apparatus comprises the step of vaporising the beer and measuring the alcohol content by the use of infra-red analysis apparatus operating in the vapour phase. As a result, the spectrum of alcohol is then distinct from the infra-red spectrum of water, and a dramatic improvement in the efficiency and accuracy of analysis is achieved. By the use of a vapour phase analysis as opposed to a liquid phase analysis as has been proposed previously, a significant improvement in performance is achieved, and the technique can be applied to analysis of the alcohol content of beer produced on a continuous basis, by arranging for sampling and vaporisation of the beer on a corresponding continuous basis.

In our published prior European patent application number EP 0 243 139 there is disclosed gas phase infra-red analysis apparatus capable of continuously measuring the concentration of up to three infra-red absorbing components in a gas phase stream. We hereby incorporate in the present application the entire disclosure of our prior European application.

In the preferred embodiment of the present invention, small volumes of beer are tapped from a main beer production pipeline. A small fixed rate of flow is defined by a pump, such as a peristaltic pump. This low rate of flow of beer is delivered to an evaporator maintained at approximately 110°C (the preferred range being from 100°C to 120°C), and in the evaporator both the water and the alcohol constituents of the beer boil. During the boiling, the increase in volume is more than one thousand times. This great increase is volume causes the vapour to force itself through a pipe kept at above 100°C into the infra-red analyser of the type disclosed in our prior

European application. The analyser has a sample cell of total path length of one metre. The path is a folded path. The beer vapour passes through the sample cell which is kept at a fixed temperature above 100°C and passes to waste.

The analyser, being a multi-component analyser, can measure water vapour content, alcohol content and carbon dioxide content simultaneously. The wavelengths of measurement of each of these are distinct, with water vapour absorbing in the region of the spectrum between 2.5 and 3.0 microns and between 5 microns and 8 microns, alcohol absorbing mainly at 3.4 microns and carbon dioxide absorbing mainly at 4.28 microns. Simple measurement at these wavelengths is sufficient to make accurate measurements of alcohol and carbon dioxide. Cross sensitivities are easily corrected-for by means of a signal processor. To zero the apparatus, distilled water is fed into the evaporator in place of beer.

In the embodiment, simple flushing can be carried out to clear away residues left by previous liquids analysed, and likewise of cleaning fluids. It is noteworthy that the carbon dioxide content of beer can be measured simultaneously with the alcohol content.

In the preferred embodiment, continuous monitoring of alcohol content is provided by the use of sampling means connected to a beer delivery pipeline. The sampling means delivers a sample to the evaporator, which puts the sampled liquid into the vapour phase, after which the infra-red or other electromagnetic radiation analyser determines quantitatively the components of the sample. Signal processing apparatus analyses the signals generated by the analyser into readable data.

Thus, according to the invention, a method of analysing the alcohol content of beer, wine etc, and applicable to the analysis of other solutions, comprises the step of causing the sample to be vaporised. Such vaporisation may be achieved by heating, or indeed by pressure reduction, or by both.

The spectrum of alcohol in vapour-phase near infra-red

analysis is distinct from the spectrum of water and other constituents such as carbon dioxide, whereby the vapour phase analysis is notably improved with respect to liquid phase analysis.

Also in the preferred embodiment, the analyser comprises datum means including a cell containing water vapour. Means is provided to prevent condensation of the water vapour during analysis. Typically, such means comprises heating means for the cell.

An embodiment of the invention will now be described by way of example with reference to the accompanying drawing which shows apparatus for the analysis of the alcohol content of beer.

In the apparatus of the drawing, the sequence of operations for analysing a sample of beer comprises the steps of removing a sample of beer through a tube 10 which taps into a main beer pipeline 12. The rate of flow of beer through tube 10 is regulated by a pump 14. The tube 10 feeds the sample into an evaporator 16 which is maintained at a temperature in the range of 105 to 115°C, preferably 110°C. The evaporator 16 is connected to a gas analysis cell 20 by means of a connecting tube 18.

Once in the evaporator 16, the sample boils and vaporises. The vaporisation causes an increase in the volume of the sample of more than one thousand-fold. This increase in volume causes the vapour to force itself into connecting tube 18 and hence into cell 20. The connecting tube 18 is maintained at a temperature above 100°C, and likewise the cell 20 of analyser 21, so as to maintain the beer in the vapour phase and prevent condensation. Temperature maintenance is by means of a temperature controller 24 controlling a heat source (not shown).

During passage of the vapour through the sample cell 20, analyser 21 carries out infra-red analysis of the vapour phase constituents of the beer namely alcohol, carbon dioxide and water. Signal processing apparatus 26 is connected to

analyser 21 and analyses signals generated thereby so as to produce readable data. The function of analyser 21 and signal processing apparatus 26 is substantially as disclosed in our prior European patent application.

Analyser 21 comprises datum means (not shown) to intercept the radiation and to transmit it to a sensor at a reference or datum value. Such datum means comprises one or more gas cells containing a relatively high concentration of gas to be analysed. In the present application, at least one of the cells of the datum means contains water vapour or steam. The temperature of the apparatus maintains the steam in the vapour phase.

Interestingly, the above embodiment provides a method and apparatus for measuring the alcohol content of wines and beers providing greater accuracy of results, as a result of the vapour phase analysis technique, and without the need to take samples for laboratory analysis. Continuous monitoring is conveniently carried out.

The method and apparatus described in the above embodiment can be used for the analysis of dissolved components in any suitable liquid, where the constituents of the solution can be vaporised and detected by an electromagnetic radiation analysis device.

Modifications which could be made in the above embodiment while remaining within the scope of the invention include the use of alternative radiation frequencies such as ultra-violet, with appropriate changes in the radiation source and the radiation sensor. A further modification comprises the provision of modified means for maintenance of the liquids in the vapour phase, including the use of reduced pressure and different ranges of temperatures.

The embodiment provides significant advantages over currently available chromatography techniques in view of the maintenance requirements of such apparatus and their cycle times.

CLAIMS :-

1 A method of analysis of liquid samples comprising :

a) providing a radiation source and employing same to transmit radiation through a sample to be analysed;

b) providing a radiation sensor and employing same to sense radiation from said source, transmitted through said sample; and

c) providing signal processing apparatus and employing same to analyse signals generated by said sensor in response to radiation transmitted to determine the content of the sample;

characterised by

d) the step of treating said liquid sample to cause it to enter the vapour phase prior to transmission of said radiation through the sample.

2 A method of analysis of liquid samples characterised by treating a liquid sample to cause it to enter the vapour phase and then subjecting the vapour phase to radiation analysis.

3 A method according to claim 1 or claim 2 characterised by providing datum means positionable to intercept radiation from said source and employing said datum means to transmit the radiation to said sensor at a reference or datum value.

4 A method according to claim 3 characterised by transmitting said radiation through a cell containing water vapour or steam.

5 A method according to claim 4 characterised by the step of maintaining said cell at a temperature and/or pressure such as to maintain said steam in the vapour phase.

6 A method according to any one of the preceding claims characterised by providing liquid samples to said apparatus

from liquid production apparatus requiring continuous or sequential monitoring of liquid content.

7 Apparatus for the analysis of liquid samples comprising:

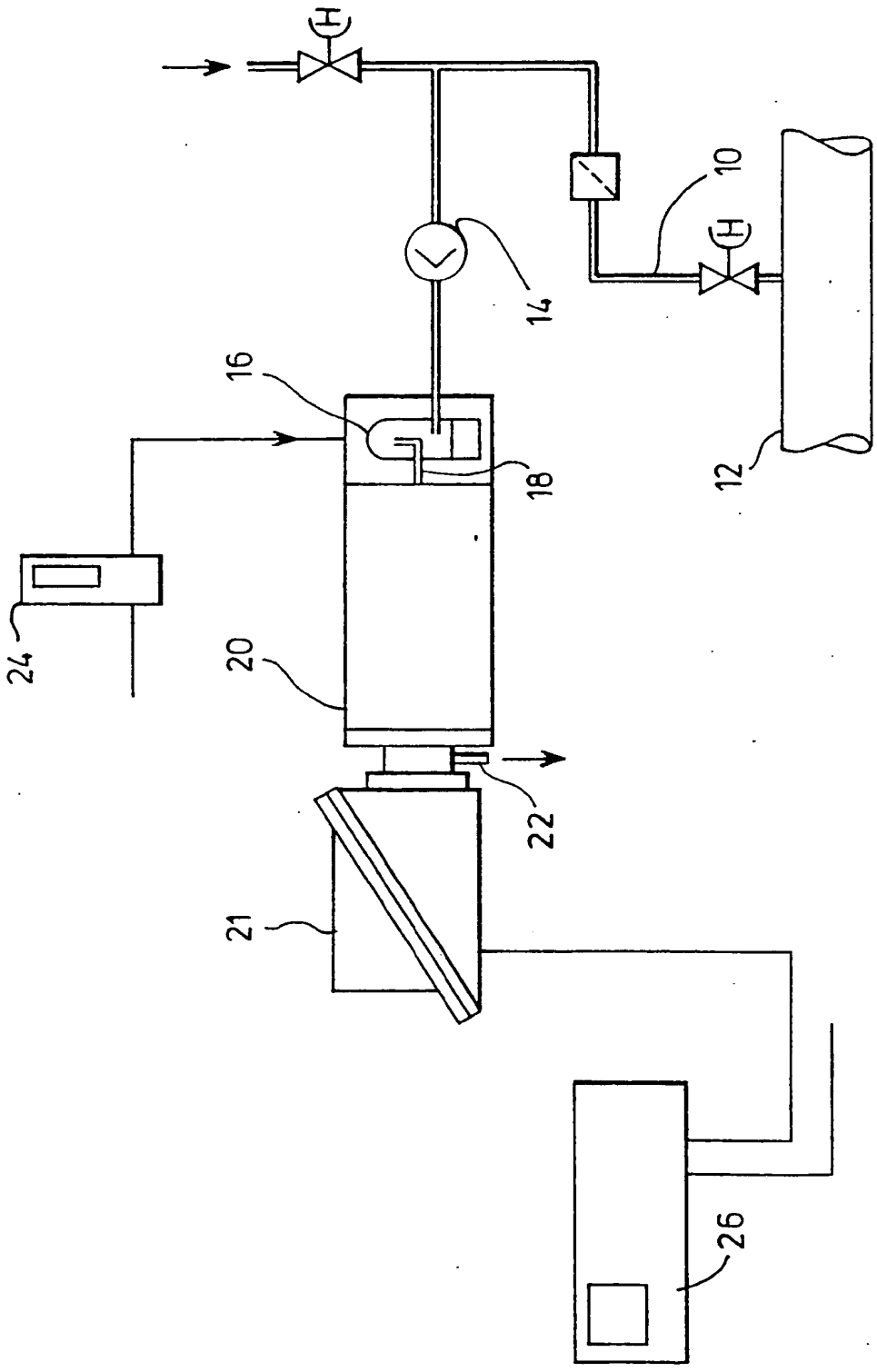
a) a gas phase analysis apparatus comprising a radiation source and a radiation sensor;

characterised by

b) means for vaporising a liquid sample to be analysed and for subjecting the resultant vapour phase to radiation from said analyser.

8 Apparatus according to claim 7 characterised by means for maintaining said liquid sample in the vapour phase.

9 Apparatus according to claim 8 characterised by said means for maintaining said sample in the vapour phase comprising heating means.



INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 93/00365

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 G01N21/35; G01N33/14

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	G01N

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
X	FR,A,670 046 (SOCIÉTÉ ANONYME DES CHARBONS ACTIFS ET PROCEDÉS ÉDOUARD URBAIN) 23 November 1929 see page 1, line 13 - line 20 see page 1, line 39 - page 2, line 8 see page 2, line 82 - line 89	1,2,7-9
Y	---	3
Y	EP,A,0 243 139 (PROCAL ANALYTICS) 28 October 1987 cited in the application see abstract; claim 1; figure 1	3
A	---	1,7
A	GB,A,2 217 838 (SHELL) 1 November 1989 see page 5, line 24 - line 33; figure 1	1,3,4

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° Special categories of cited documents :[10]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
27 MAY 1993	18. 06. 93
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	KRAMETZ E.M.

Form PCT/ISA/210 (second sheet) (January 1985)

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	DE,A,2 809 910 (DIESSEL) 22 March 1979 see claims 1,2	1,7

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

GB 9300365
SA 70977

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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EP-A-0243139	28-10-87	JP-A- 63012938	20-01-88
GB-A-2217838	01-11-89	None	
DE-A-2809910	22-03-79	GB-A,B 2018423 US-A- 4228192	17-10-79 14-10-80

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82